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Emission Measurements of Ultracell XX25 Reformed Methanol Fuel Cell System

by Charles Rong, Dat Tran, Elizabeth Ferry, and Deryn Chu

ARL-TR-4477 **June 2008**

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14. ABSTRACT
Four Ultracell XX25 Rev B. Reforming Methanol Fuel Cell systems have been tested for exhaust emissions at the U.S. Army Research Laboratory (ARL) under different operating conditions. These tests were conducted at a government facility to verify data previous reported and measured at Ultracell facilities. Two identical Agilent micro-GC systems were used to carry out the test. Personnel from Ultracell Corporation and Agilent Technologies, and Government Personnel from CERDEC and ARL participated in the test. This report documents the test procedure, analytical instrument calibration, operating parameters, and the obtained results. Hazardous exhaust emission components, such as carbon monoxide, methane, and methanol, were found during the start up time, and more found during the cold start up time (the fuel cell system was pre-soaked at 0 ºC for 1.5 h). However, all emissions that were detected and reported in this document are expected to be below the OSHA standards for exposure limits if the system is subjected to the OSHA conditions for measurements. The cause of the emission was the formation of some incompletely burned methanol molecule that was used to heat up the reformer and fuel cell stack in the system. The efficiency and reliability of the burner over the life time of the fuel cell system is at the center of the issue, a common understanding accepted by all the parties involved in the testing.

15. SUBJECT TERMS
Methanol, Reforming, Fuel Cell, Emission, Carbon Monoxide, Carbon Dioxide, Formaldehyde

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1. Introduction

Research and development of reformed methanol fuel cell (RMFC) has been active recently (*1 through 3*) for its higher temperature operation for better carbon monoxide (CO) tolerance and improved electrochemical kinetics. The Army needs high-energy density, light-weight power sources for the dismounted warrior and this demand for electric power in battlefield will be best met by high energy content liquid fuels through the combination of electrochemical devices such as fuel cell and battery. Polymer electrolyte membrane fuel cells (PEMFC) using hydrogen or liquid hydrocarbons, such as alcohols, as fuel are candidates--along with batteries and capacitors--to fill the power needs for advanced weapons, communications and computers, sensors and night vision capabilities.

High temperature PEMFC offers some advantages such as enhanced electrode kinetics and better tolerance of carbon monoxide that will poison the conventional PEMFC. Ultracell Corporation, Livermore, California has developed a first generation of 25 W RMFC System XX25 for portable Soldier power generation sponsored by the Army over the last several years. U.S. Army Communications-Electronics Research, Development and Engineering Center's (CERDEC) Army Power Division has found through laboratory tests the Ultracell XX25 system to be the most advanced and mature fuel cell system to date. Last year, CERDEC coordinated with the Joint Readiness Training Center at Fort Polk, LA (JRTC) to conduct the first of its kind Soldier field test of a fuel cell system being operated in an actual training scenario.

Unlike a direct methanol fuel cell (DMFC) where methanol fuel is directly sent to the anode for fuel cell operation, in the RMFC the methanol fuel is first reformed into hydrogen and carbon monoxide and the reformate is then sent to the anode of the fuel cell. Since carbon monoxide is produced during the operation of the fuel cell, and the system burns methanol fuel in order to heat up the reformer and the fuel cell, it is important to gain an understanding of the exhaust composition from the RMFC under various operating conditions for both the safety of the end user and for the improvement of the technology.

At the request of Program Manager, Soldier Warrior (PM SWAR), CERDEC, and U.S. Army Research Office (ARO), the emission test was first conducted at Ultracell facilities from January 28 to February 1, 2008. Nondispersive infrared sensor (NDIR) and gas chromatograph (GC) were used to measure CO, CO_2, and methanol exhaust emissions. A detailed documentation of the test completed at Ultracell is available as an internal report (*4*). To provide government verification of the results obtained at Ultracell, a second emission test of the XX25 RMFC was conducted at the U.S. Army Research Laboratory, Adelphi, Maryland from March 17 to 21, 2008. This report documents the procedure and results of the emission test conducted at Adelphi. All the authors of this report participated in the testing. Other participants were Gerry Tucker and Frank Chan from Ultracell and Vince Giarrocco from Agilent Technologies.

2. Experimental

2.1 Analytical Instrument

Agilent Micro-GC was used to measure the concentrations of carbon monoxide, carbon dioxide, and methane from the fuel cell exhaust. Formaldehyde and methanol were measured qualitatively. Three channels were used: the first one for methane and carbon monoxide, the second for carbon dioxide, and the third for formaldehyde and methanol. The first channel column (labeled A, molecular sieve) was pre-heated/conditioned at 180 ºC for 48 h before the measurements. Each sample run took about 3 min and two identical Micro-GC's were used to make measurements separated by one and a half minute interval, that is, after the first Micro-GC had run for one and a half minutes, the second Micro-GC started taking sample. This way we can obtain data point at the rate of every one and a half minute instead of every three minute with one Micro-GC. The instrument setups are shown in figures 1, 2, and 3. Analytical method parameters are listed in table 1.

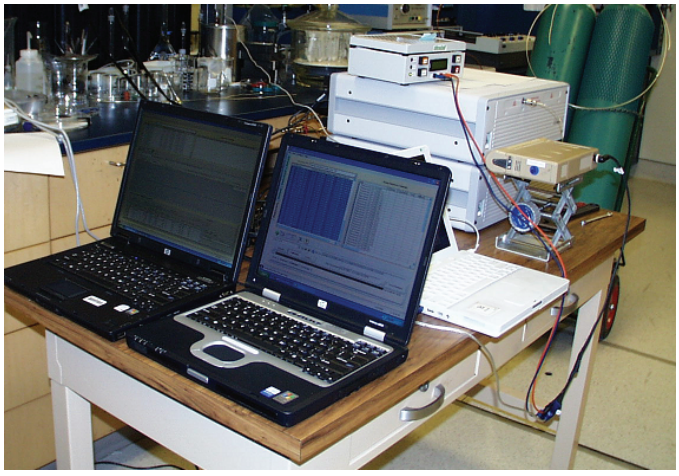

Figure 1. Two Micro-GC systems set up for the measurements.

Figure 2. Ultracell XX25 system was placed horizontally next to the Micro-GC.

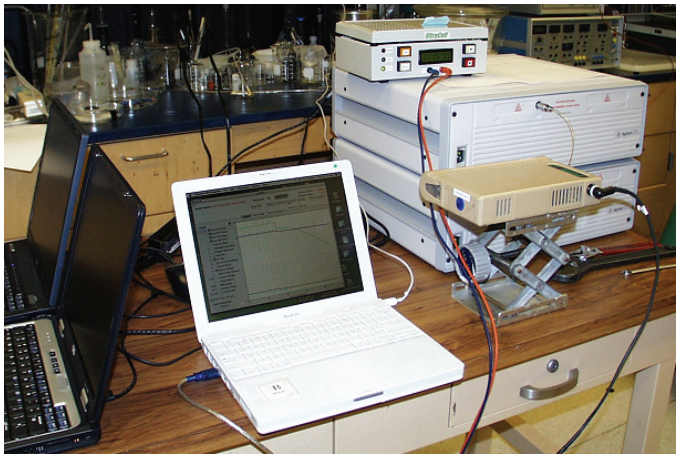

Figure 3. The electrical load device controlled by a notebook located on the top of the Micro-GC.

Table 1. Parameters of Micro-GC applied during the measurements.

Channel	**A**	**(Molecular Sieve)**	**B**	**(Plot U)**	**D**	**(OV-1)**
Sample Inlet Temp.	On	100 ºC	On	100 ºC	On	100 ºC
Injector Temp.	On	90 ºC	On	90 ºC	On	90 ºC
Column Temp.	On	90 ºC	On	80 ºC	On	52 ºC
Sample Pump	On	20 s	On	20 s	On	20 s
Inject Time		50 ms		50 ms		40 ms
Run Time		132 s		90 s		90 s
Post Run Time		0 s		0 s		0 s
Pressure Equilibrium Time		0 s		0 s		0 s
Column Pressure	On	35.00 psi	On	32.00 psi	On	25.00 psi
Post Run Pressure		35.00 psi		32.00 psi		25.00 psi

Table 1. Parameters of Micro-GC applied during the measurements (continued).

Channel	A	(Molecular Sieve)	B	(Plot U)	D	(OV-1)
Detector Filament	On		On		On	
Detector Sensitivity	High		High		High	
Detector Data Rate		50 Hz		50 Hz		50 Hz
Baseline Offset		0 mV		0 mV		0 mV
Backflush Time		12.0 s		6.0 s		~

2.2 Instrument Calibration

Two certified calibration gas mixtures were used to calibrate the Micro-GC for methane, carbon monoxide, and carbon dioxide. The gases were from Scott Specialty Gases with concentrations listed in table 2.

Table 2. Concentration of calibration gas mixture (ppm).

	CH_4	CO	CO_2	
No. 1	101	501	1,000	Balanced by N_2
No. 2	502	1,000	10,000	Balanced by N_2

2.3 Measurements of the Fuel Cell

A total of four Ultracell XX25 units were tested under different operating condition as outlined in the Results and Discussion section. Each running profile consists of operation of start-up for 20 min; at half load level of 12.5 W for 15 min; at the full power level of 25 W for 15 min; at idling condition (no load) for 10 to 15 min, and finally during shut down process for about 10 min. Tests were first completed at room temperature. Further tests were conducted when the fuel cell systems were pre-conditioned at 0 ºC for 1.5 h and 50 ºC for 3 h and then immediately turned on for operation at room temperature.

3. Results and Discussion

3.1 Measurements at Room Temperature

The measurements of RMFC system under horizontal condition started at room temperature without any pre-treatment in temperature were conducted as follows: start-up for 20 min; at half load level of 12.5 W operation for 15 min; at the full power level of 25 W for 15 min; at idling condition (no load) for 10 to 15 min, and finally during shut down process for about 10 min. All four units were tested using this method. Figure 4a-4d show the quantitative data during one complete testing cycle for one of the four units and table 3 summarizes the average data point during the start-up 20 min as well as the results of qualitative examination of the chromatographic data for any formaldehyde and methanol.

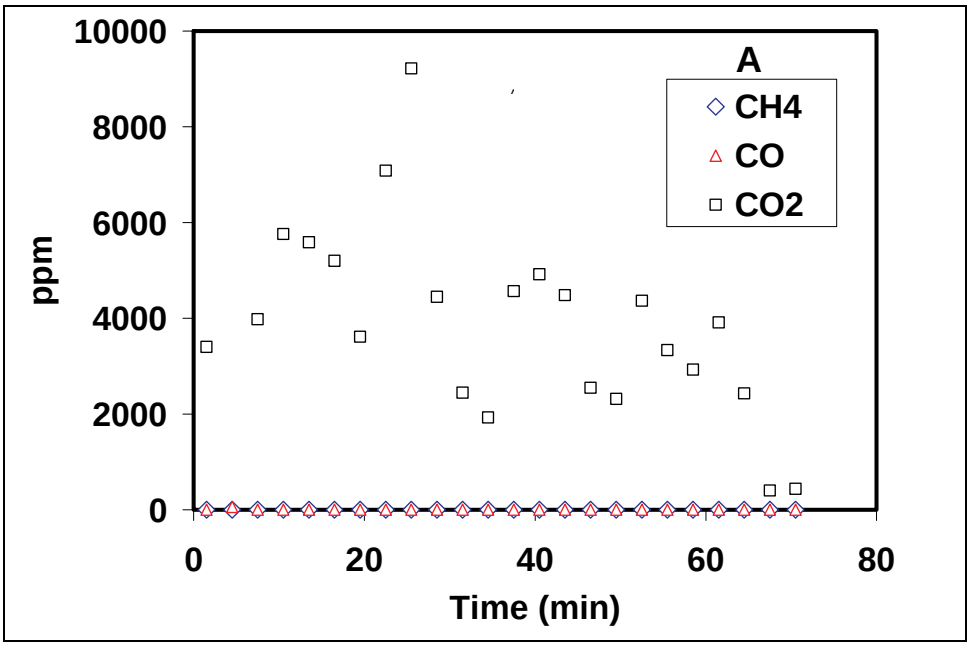

Figure 4a. XX25 Unit Two exhaust composition started from room temperature measured by Micro-GC 1. The operation includes start-up for 20 min; at half load level of 12.5 W operation for 15 min; at the full power level of 25 W for 15 min; at idling condition (no load) for 10-15 min, and finally during shut down process for about 10 min with total running time for more than 70 min.

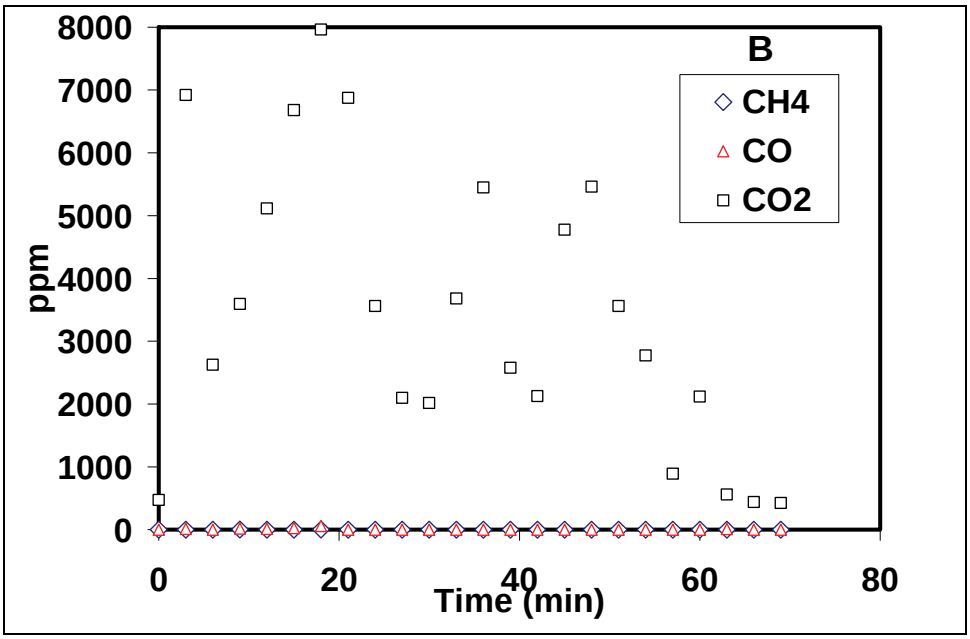

Figure 4b. XX25 Unit Two exhaust composition started from room temperature measured by Micro-GC 2. The operation includes start-up for 20 min; at half load level of 12.5 W operation for 15 min; at the full power level of 25 W for 15 min; at idling condition (no load) for 10-15 min, and finally during shut down process for about 10 min with total running time for more than 70 min.

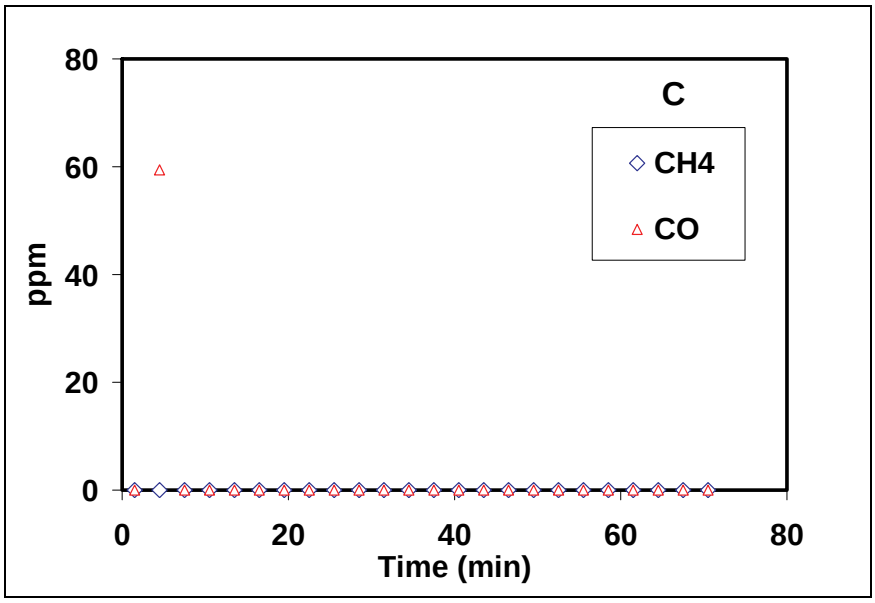

Figure 4c. XX25 Unit Two exhaust composition started from room temperature measured by Micro-GC 1. The operation includes start-up for 20 min; at half load level of 12.5 W operation for 15 min; at the full power level of 25 W for 15 min; at idling condition (no load) for 10-15 min, and finally during shut down process for about 10 min with total running time for more than 70 min.

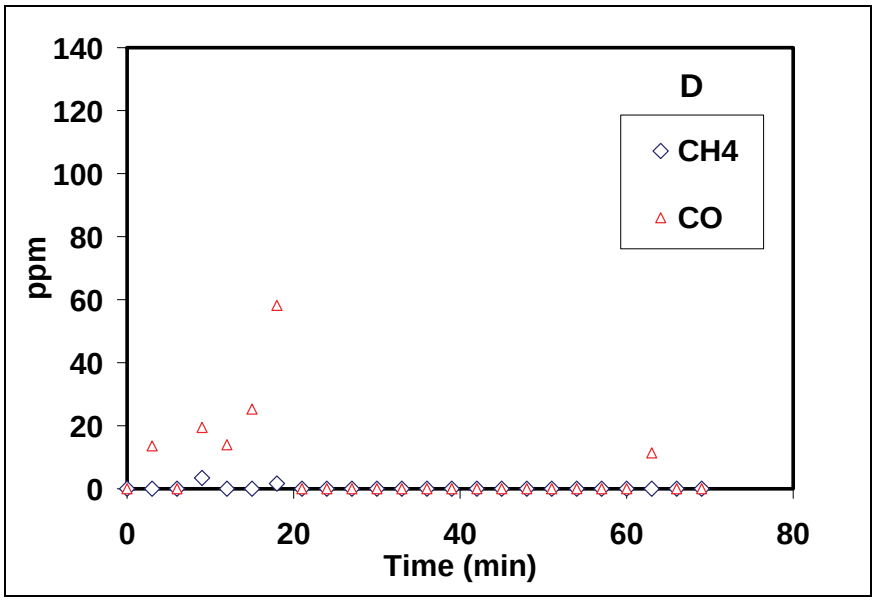

Figure 4d. XX25 Unit Two exhaust composition started from room temperature measured by Micro-GC 2. The operation includes start-up for 20 min; at half load level of 12.5 W operation for 15 min; at the full power level of 25 W for 15 min; at idling condition (no load) for 10-15 min, and finally during shut down process for about 10 min with total running time for more than 70 min.

Table 3. Summary of the average data point for the start-up time (20 min) of the four units that were tested. N/D: not detected.

	CO (PPM)	**CO_2 (PPM)**	**CH_3OH**	**CH_4 (PPM)**	**HCHO**
Unit One	~78	~5257	Trace	~40	N/D
Unit Two	~32	~4766	Trace	~3	N/D
Unit Three	~23	~5130	Trace	~5	N/D
Unit Four	~38	~5206	Trace	~10	N/D

Figures 4a and 4b show that the Unit Two emitted carbon dioxide during the run time with no obvious pattern from both of the Micro-GCs. The start-up time operation seemed to be producing more carbon dioxide than operation under load when the system has reached the desired temperature. Table 3 summarizes the averaged data point for the four units ranging from 4800 to 5300 ppm CO_2. However, carbon monoxide formation and release was only found during the start up 20 min, which was on average from 20 to 80 ppm, see figure 4c and 4d. Note each of the Micro-GC took sample in every three minute so that the data shown in table 3 may not represent a constant release of carbon monoxide at such level. The second Micro-GC, expected to perform very closely but not identically, played a compensating role to provide an average sampling at one and a half minute interval and we observed no unexpected results. Methanol and methane were only found during the start up time, along with carbon monoxide. However, in all cases the emission levels measured with the micro-GC were mostly below the OSHA standards for exposure to these gases, see table 4. No formaldehyde was found for any of the units. The emission during the start up is the main concern as this is the time when the fuel cell system would be burning methanol fuel directly to heat up the system for operation. The test results further confirmed and verified the previous conclusions made by Ultracell. From system to system, there is a variation of the emission data as shown in table 3. If this variation is determined to be significant, Ultracell will address it in its manufacturing process in the future.

Table 4. OSHA Chemical Exposure Limits (*5*).

Compound	PEL TWA 8h	Fatal Exposure	Overexposure Symptoms	Miscellaneous
Carbon Monoxide	50 ppm	800 ppm/1h 300 ppm/2h	Flu-like symptoms (low) Dizzy, drowsy (medium) Unconsciousness, brain damage (high)	Colorless, odorless, tasteless gas
Carbon Dioxide	5000 ppm	Asphyxiation	Headache, dizzy, sweating, convulsion	Colorless, odorless
Formaldehyde	0.75 ppm 2 ppm for 15 min		Eye irritant, skin burns, respiratory irritant, headache, dizzy, nausea, chest pain	Also absorbed through skin
Formic Acid	5 ppm		Corrosive to eyes, burns to respiratory tract, skin burns	
Methane	None	Asphyxiation	Headache, dizzy, vomiting, convulsion	
Methanol	200 ppm	100 to 125 ml	Impaired vision, nausea, headache, dizzy	Also absorbed through skin; indirect poison: breaks down in liver to form formic acid and formaldehyde

PEL: permitted exposure limit; TWA: time weighted average.

3.2 Measurements After Pre-conditioned at 0 ºC for 1.5 h

The measurements of RMFC system that was pre-soaked at 0 ºC for 1.5 h and then immediately tested at room temperature under upright condition were conducted as follows: start-up for 20 min; at half load level of 12.5 W operation for 15 min; at the full power level of 25 W for 15 min; at idling condition (no load) for 10-15 min, and finally during shut down process for about 10 min. Two of the four units (Unit One and Two) were tested in this fashion.

Carbon dioxide release for this cold start test from 0 ºC seems to be not much different than the room temperature start up test, see figure 5a and 5b. However, as shown in figure 5c and 5d, we found generally more of data points representing the carbon monoxide and methane formation and release that extended to the operation under full load. This suggests that the system may not reach an equilibrium temperature after 20 minute running from 0 ºC pre-soaking condition. Tables 5 and 6 summarized the average data points during the start up and full load operations.

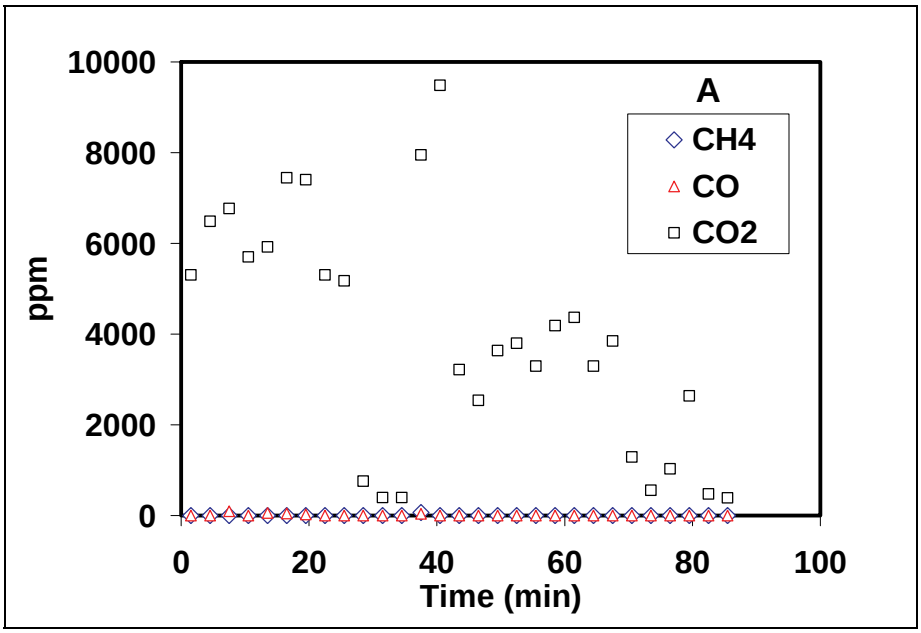

Figure 5a. XX25 Unit Two exhaust composition measured by Micro-GC1. The unit was pre-soaked at 0 ºC for 1.5 h and immediately started at room temperature. The operation profile is start-up for 20 min; at half load level of 12.5 W operation for 15 min; at the full power level of 25 W for 15 min; at idling condition (no load) for 10-15 min, and finally during shut down process for about 10 min with total running time for more than 70 min.

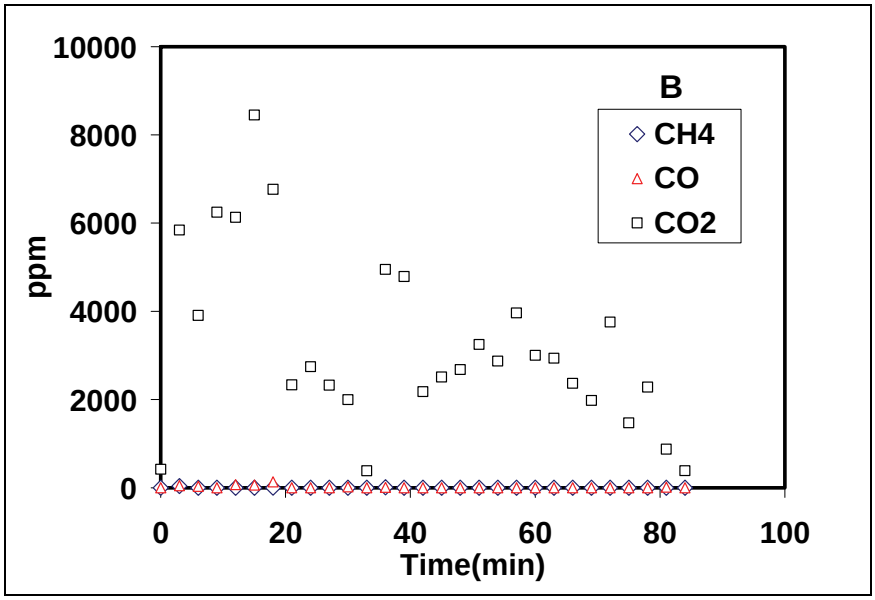

Figure 5b. XX25 Unit Two exhaust composition measured by Micro-GC2. The unit was pre-soaked at 0 ºC for 1.5 h and immediately started at room temperature. The operation profile is start-up for 20 min; at half load level of 12.5 W operation for 15 min; at the full power level of 25 W for 15 min; at idling condition (no load) for 10-15 min, and finally during shut down process for about 10 min with total running time for more than 70 min.

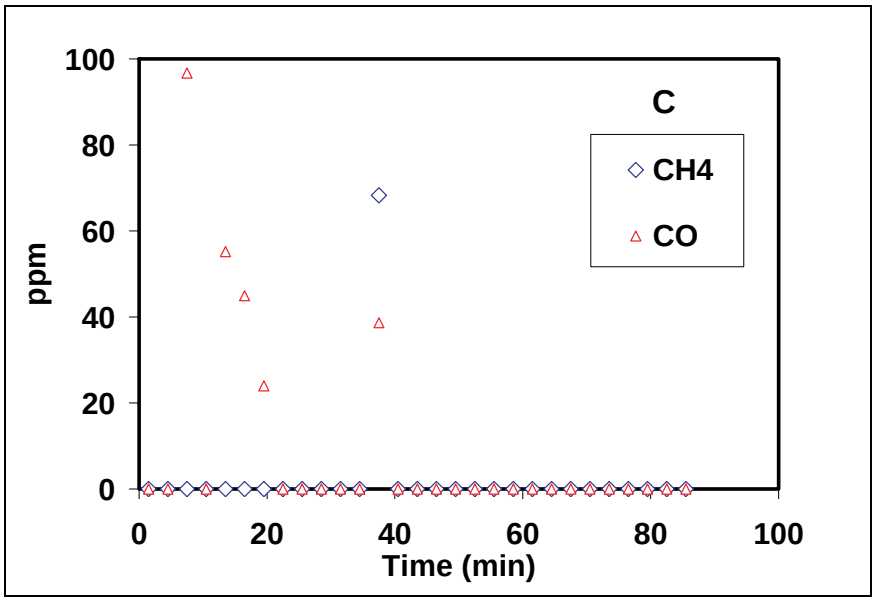

Figure 5c. XX25 Unit Two exhaust composition measured by Micro-GC1. The unit was pre-soaked at 0 ºC for 1.5 h and immediately started at room temperature. The operation profile is start-up for 20 min; at half load level of 12.5 W operation for 15 min; at the full power level of 25 W for 15 min; at idling condition (no load) for 10-15 min, and finally during shut down process for about 10 min with total running time for more than 70 min.

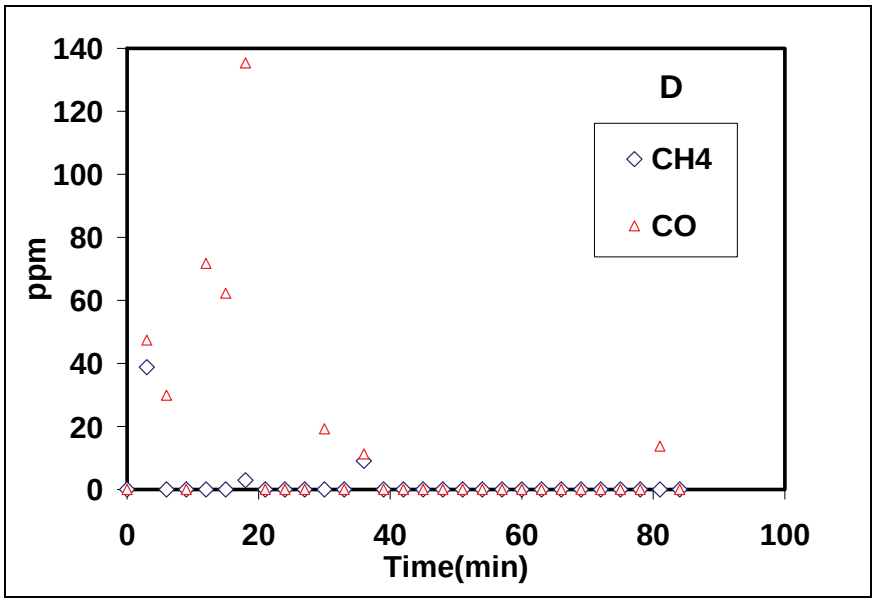

Figure 5d. XX25 Unit Two exhaust composition measured by Micro-GC2. The unit was pre-soaked at 0 ºC for 1.5 h and immediately started at room temperature. The operation profile is start-up for 20 min; at half load level of 12.5 W operation for 15 min; at the full power level of 25 W for 15 min; at idling condition (no load) for 10-15 min, and finally during shut down process for about 10 min with total running time for more than 70 min.

Table 5. Summary of the average data point for the start-up time (20 min) of the Unit One and Two after pre-soaked at 0 ºC for 1.5 h. N/D: not detected.

	CO (PPM)	**CO_2 (PPM)**	**CH_3OH**	**CH_4 (PPM)**	**HCHO**
Unit One (0 °C)	~33	~4072	Trace	N/D	N/D
Unit Two (0 °C)	~63	~5394	Trace	~22	N/D

Table 6. Summary of the average data point for the full load operation at 25 W (15 min) of the Unit One and Two after pre-soaked at 0 ºC for 1.5 h. N/D: not detected.

	CO (PPM)	**CO_2 (PPM)**	**CH_3OH**	**CH_4 (PPM)**	**HCHO**
Unit One (0 °C)	~17	~4651	Trace	Trace	N/D
Unit Two (0 °C)	~23	~3046	Trace	~38	N/D

3.3 Measurements After Pre-Conditioned at 50 ºC for 3 h

The measurements of RMFC system that was pre-soaked at 50 ºC for 3h and then immediately tested at room temperature under upright condition were conducted as follows: start-up for 20 min; at half load level of 12.5 W operation for 15 min; at the full power level of 25 W for 15 min; at idling condition (no load) for 10-15 min, and finally during shut down process for about 10 min. Two others of the four units (Unit Three and Four) were tested in this fashion.

The 50 ºC pre-heating of the system had a positive impact in terms of reduced emission of those undesired gases. Only a couple of data points were shown in figures 6a and 6b during the start up running of 20 min and nothing was observed afterward. Started at 50 ºC, the burner consumed less methanol fuel to heat up the reformer and fuel cell stack, and as a result, less carbon monoxide and methane were generated and released in the exhaust. Table 7 summerized the average data points for this experiment. The key issue to be addressed immediately is to further improve the efficiency of the burner to completely convert methanol fuel to carbon dioxide and water. It is known that a catalyst component has been installed in the system and the test results in this report suggest that the operation parameters and its control software adjustment may help remedy the problem.

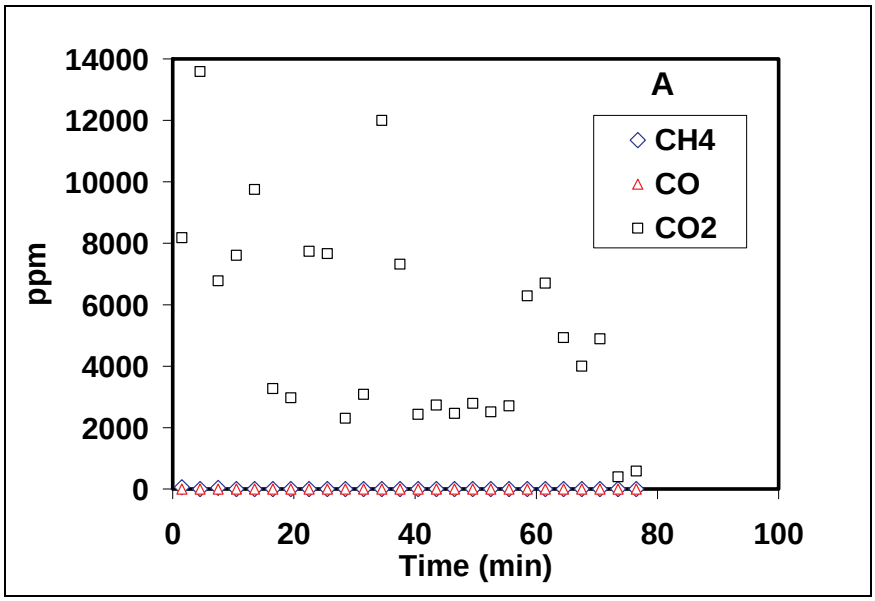

Figure 6a. XX25 Unit Four exhaust composition measured by Micro-GC1. The unit was pre-soaked at 50 ºC for 3 h and immediately started at room temperature. The operation profile is start-up for 20 min; at half load level of 12.5 W operation for 15 min; at the full power level of 25 W for 15 min; at idling condition (no load) for 10-15 min, and finally at shut down process for about 10 min with total running time for more than 70 min.

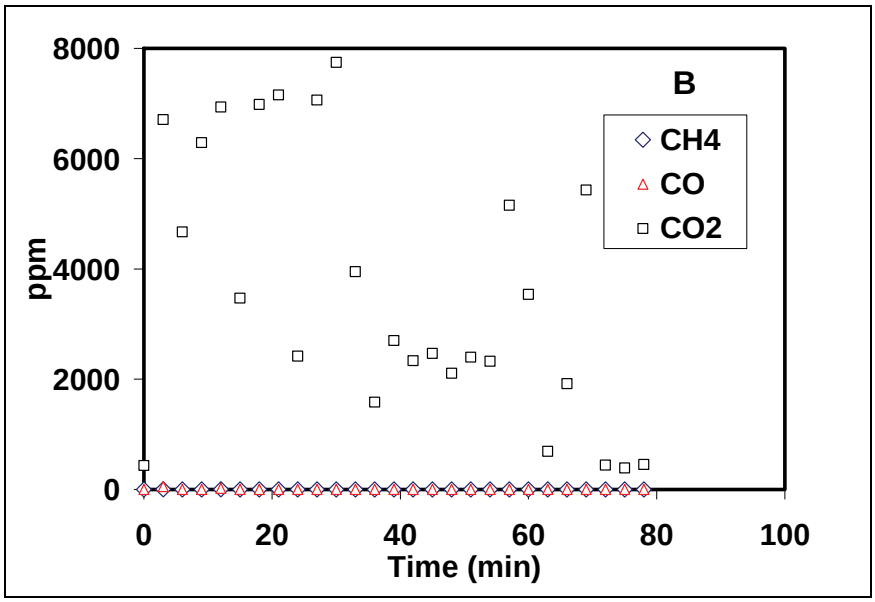

Figure 6b. XX25 Unit Four exhaust composition measured by Micro-GC2. The unit was pre-soaked at 50 ºC for 3 h and immediately started at room temperature. The operation profile is start-up for 20 min; at half load level of 12.5 W operation for 15 min; at the full power level of 25 W for 15 min; at idling condition (no load) for 10-15 min, and finally at shut down process for about 10 min with total running time for more than 70 min.

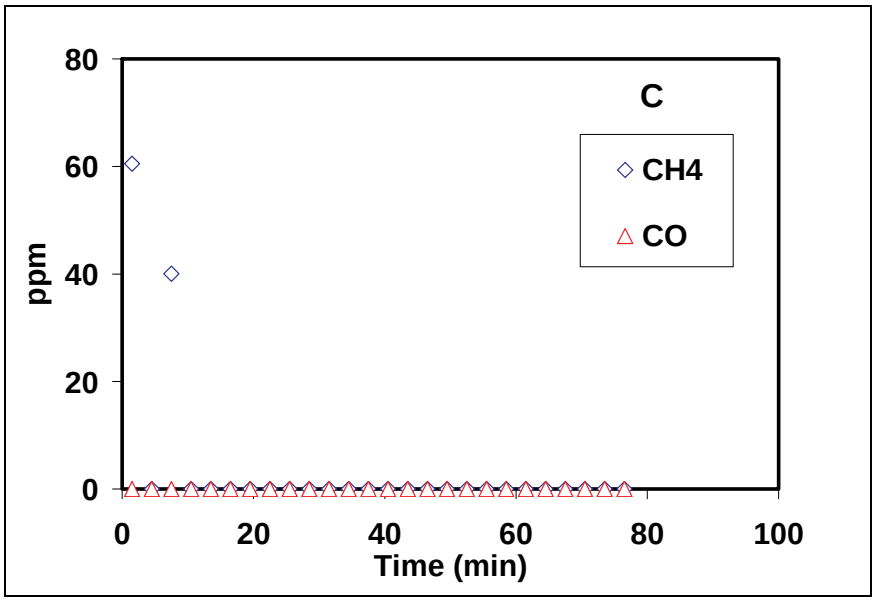

Figure 6c. XX25 Unit Four exhaust composition measured by Micro-GC1. The unit was pre-soaked at 50 ºC for 3 h and immediately started at room temperature. The operation profile is start-up for 20 min; at half load level of 12.5 W operation for 15 min; at the full power level of 25 W for 15 min; at idling condition (no load) for 10-15 min, and finally at shut down process for about 10 min with total running time for more than 70 min.

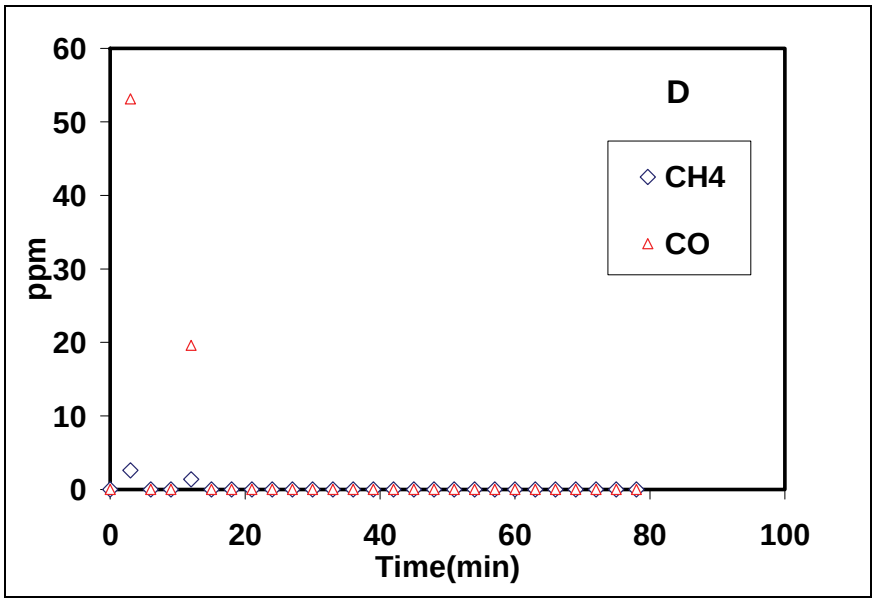

Figure 6d. XX25 Unit Four exhaust composition measured by Micro-GC2. The unit was pre-soaked at 50 ºC for 3 h and immediately started at room temperature. The operation profile is start-up for 20 min; at half load level of 12.5 W operation for 15 min; at the full power level of 25 W for 15 min; at idling condition (no load) for 10-15 min, and finally at shut down process for about 10 min with total running time for more than 70 min.

Table 7. Summary of the average data point for the start-up time (20 min) of Unit Three and Four after pre-soaked at 50 ºC for 3 h. N/D: not detected.

	CO (PPM)	**CO_2 (PPM)**	**CH_3OH**	**CH_4 (PPM)**	**HCHO**
Unit Three (50 °C)	~22	~4726	Trace	~25	N/D
Unit Four (50 °C)	~36	~6091	Trace	~26	N/D

4. Conclusion

The experimental results from the four units of Ultracell XX25 Reforming Methanol Fuel Cell system tested at ARL facility showed that the carbon monoxide, methane, and methanol in the exhaust were basically below all OSHA exposure limits for these gases during the start up stage and at virtually zero level when the system reached normal operating temperature. The system started from pre-soaked condition at 0 ºC generated slightly more of the carbon monoxide, methane, and methanol than that started from room temperature as a result that more methanol fuel was needed to heat up the system. However, even under these circumstances, the XX25 emissions levels are expected to be below the permitted exposure limits for these gases under OSHA conditions. On the other hand, when the system started at 50 ºC, it generated less of the carbon monoxide, methane, and methanol. The cause of these undesired emissions was some incompletely burned methanol molecules that are used to heat up the reformer and fuel cell stack in the system. The efficiency and reliability of the burner over the lifetime of the fuel cell system is at the center of the issue, a common understanding accepted by all the parties involved in the testing.

5. References

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